IN THE UNITED STATES

PATENT AND TRADEMARK OFFICE

PATENT APPLICATION

INVENTORS: Sang-Wook Cheong
Namjung Hur

CASE: 5-1

Serial No.: 09/885,471

Group Art Unit: 1762

Filed: June 20, 2001

Title: MgB_2 Superconductors

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

SIR:

DECLARATION UNDER 37 C.F.R. 1.131

1. I, Sang-Wook Cheong, received a physics Ph.D. in 1989 from the University of California at Los Angeles. I am presently a Professor of Physics at Rutgers University in Piscataway, NJ. I was a member of the technical staff at the Bell Laboratories of Lucent Technologies Inc, Murray Hill NJ, for more than 10 years. At Bell Laboratories, I did research and published research articles in: superconductivity, magnetism, and materials synthesis.

2. I, Namjung Hur, am a graduate student in physics at Rutgers University in Piscataway, NJ.

We the Declarants, Sang-Wook Cheong and Namjung Hur, state further that:

3. We are both inventors on, at least, some of pending claims 8 – 15 in the above application and on, at least, some of originally filed claims 1 – 7 in the above-referenced patent application.

4. Prior to March 2001, we formulated ideas for processes to grow a MgB_2 layer and for structures having a MgB_2 layer.

Cheong 5-1

5. Our formulation of the ideas in above paragraph 4 is corroborated by an entry at page 23 of a notebook where Dr. Cheong recorded new discoveries. Dr. Cheong prepared, signed, and dated the entry at page 23 of the notebook prior to March 2001. A copy of said page is attached as Exhibit 1. In Exhibit 1, the date of Dr. Cheong's signature and a date on the upper left hand portion of the notebook page have been redacted. The page of the notebook also includes a signature of another person who read the page after March 8, 2001.

6. Prior to March 2001, our ideas for growing a MgB_2 layer included the following process steps:

a) providing a solid pellet of MgB_2;

b) ejecting MgB_2 from the pellet by directing laser light thereon; and

c) growing a MgB_2 layer on a substrate's surface from the ejected MgB_2.

Exhibit 1 includes statements corroborating that our ideas included each of these steps. With respect to step (a), the notebook page of Exhibit 1 states:

> "MgB_2 powder can be purchased .... However, well-sintered, strong pellet is difficult to prepare, but the synthesis by using high-pressure and high temperature apparatus should be possible."

With respect to step (b), the notebook page of Exhibit 1 states:

> "The well-sintered pellet can be used as a target for the PLD (Pulsed Laser Deposit) film preparation."

With respect to step (c), the notebook page of Exhibit 1 states:

> "the PLD film growth may not require ultra-high vacuum, which is costly. Since the in-plane lattice constant of MgB_2 is about 3.085 Å, substrates such as ... can be utilized for the (epitaxial) film growth .... The epitaxial MgB_2 films can be utilized ..."

7. Prior to March 2001, our ideas for the processes of above paragraph 6 also included using substrates whose in-plane lattice constants match those of MgB_2 to 10 percent or better. Exhibit 1 includes a statement corroborating this. The statement proposes using 6H – SiC as a substrate and gives the associated lattice constant as a = 3.085 Å. This value matches the in-plane lattice constant of MgB_2 to better than 1 percent.

8. Prior to March 2001, our ideas for the processes of above paragraph 6 also included using a sintering process to make the solid pellet of MgB_2. The notebook page of Exhibit 1 corroborates this in the following statement:

> "MgB_2 powder can be purchased .... However, well-sintered, strong pellet is difficult to prepare, but the synthesis by using high-pressure and high temperature apparatus should be possible."

9. Prior to March 2001, our ideas for the processes of above paragraph 6 also included using a pulsed laser to eject MgB_2 from the pellet. The notebook page of Exhibit 1 corroborates this in the following statement:

> "The well-sintered pellet can be used as a target for the PLD (Pulsed Laser Deposit) film preparation."

10. Prior to March 2001, our ideas for the processes of above paragraph 6 also included using exemplary substrates such as: SiC, $LaAlO_3$, SiO_2, $SrTiO_3$, and sapphire. The notebook page of Exhibit 1 corroborates this in the following statement:

> "substrates such as 6H – SiC ..., cubic – SiC, $LaAlO_3$, sapphire and $SrTiO_3$ can be utilized for the (epitaxial) film growth."

11. Shortly after our formulation of the ideas for the processes and structures of above paragraphs 4 - 10, Dr. Cheong contacted patent attorney, John McCabe, to initiate filing of a patent application for said processes and structures. Dr. Cheong gave a copy of the notebook page of above paragraph 5 to Mr. McCabe. Dr. Cheong discussed said notebook page with Mr. McCabe in detail to help him to prepare a patent application.

12. Dr. Cheong discussed the inventions of above paragraphs 4 and 6 with Mr. McCabe, at least, once during the period between March 8, 2001 and March 12, 2001 in order to help in the filing of a patent application thereon.

13. Prior to March 2001, Namjung Hur started experiments whose goal was obtaining a solid pellet of MgB_2 for use in practicing the processes of above paragraph 6. In the experiments prior to March 2001, Mr. Hur prepared powder sample mixtures and attempted to produce solid objects of MgB_2 by sintering the sample mixtures. A "Sample Log Notebook" describes the

sample mixtures for the experiments, and a "Machine Log Notebook" describes the experimental conditions for Mr. Hur's attempts to make solid MgB_2 objects by sintering the sample mixtures.

14. Before, during, or shortly after each experiment of above paragraph 13, Mr. Hur recorded information on the experiment and the date of the experiment in the Sample and/or Machine Log Notebook. Exhibit 2 is a copy of a page of Mr. Hur's "Sample Log Notebook" with entries that record compositions and preparation conditions for powder sample mixtures NH93 and NH94. Exhibit 3 is a copy of a page of Mr. Hur's "Machine Log Notebook" with an entry that records sintering conditions used to produce a MgB_2 object labeled BB145 from the NH93 mixture. These pages of the Sample and Machine Log Notebooks show dates prior to March 2001. The dates are redacted in Exhibits 2 and 3.

15. Mr. Hur continued into March of 2001 the experiments whose goal was the obtainment of solid MgB_2 pellets for use in the processes of above paragraph 6. Before, during, or shortly after each such experiment, Mr. Hur recorded detailed information on the experiment and the date of the experiment by an entry in one or both of his Log Notebooks. Exhibit 5 is a copy of one such entry from a page of Mr. Hur's Machine Log Notebook. The entry lists details of experiments described in this paragraph. The experiments were performed on March 7, 2001 to make MgB_2 objects labeled sample BB150a and sample BB150b.

16. Herein, we certify that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true. We also understand that willful false statements and the like are punishable by fine, imprisonment or both under 18 U.S.C. 1001 and that willful false statements and the like may jeopardize the validity of the application-at-issue or any patent issuing thereon.

Date: Aug. 28, 2003

Sang-Wook Cheong

Date: 28 Aug., 2003

Namjung Hur

[redacted]

MgB_2 was discovered to be superconducting at 39 K by Akimitsu's group. MgB_2 powder can be purchased from chemical companies such as Alfaeran, and also can be synthesized readily by chemical reaction between Mg and B powder at 800-950°C. However, well-sintered, strong pellet is difficult to prepare, but the synthesis by using high-pressure and high-temperature apparatus should be possible. The well-sintered materials can be utilized to fabricate various devices such as Liquid He gauge. The well-sintered pellet can be used as a target for the PLD (Pulsed Laser Deposit) film preparation. We found that MgB_2 powders decompose only slowly in air at temperatures such as 920°C. Thus, the PLD film growth may not require ultra-high vacuum, which is rarely. Since the in-plane lattice constant of MgB_2 is about 3.085 Å, substrates such as 6H-SiC (a = 3.081 Å), cubic-SiC, $LaAlO_3$, sapphire and $SrTiO_3$ can be utilized for the (epitaxial) film growth. One might be able to grow films on flexible substrates such as nylon to produce flexible, superconducting objects. The epitaxial MgB_2 films can be utilized for various superconducting devices such as SQUID devices. SiC may be the best candidates of substrates for the epitaxial film growth.

Sang-W. Cheong [redacted]

Read & Understood

[redacted] 5/7/2001

[redacted]

Isotope ^{11}B

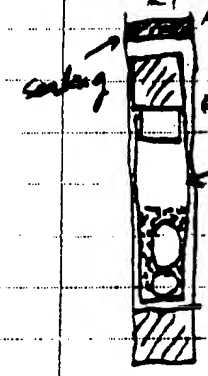
NH93 MgB_2 → actual $Mg_{1.038}B_2$

Mg	24.312	1.02	(1.1734)
B	10.811	2	1.0231

$B \rightarrow \frac{10.811}{24.312} \times \frac{2}{1.02} \times (\quad) = 0.87192 \times (1.1734)$.

LF1700

RT $\xrightarrow{100°C/h}$ 600°C $\xrightarrow{1h}$ $\xrightarrow{100°C/h}$ 800°C $\xrightarrow{1h}$ $\xrightarrow{100°C/h}$ 920°C $\xrightarrow{1h}$ $\xrightarrow{100°C/h}$ RT



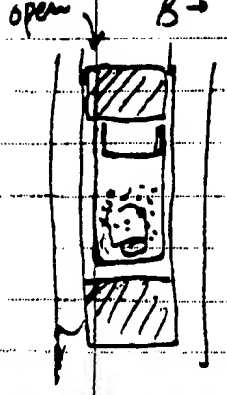
→ High pressure

Isotop ^{11}B

NH94 MgB_2 flux growth

Mg	24.312	2	(1.8293)
B	10.811	1	0.4067

$B \rightarrow \frac{10.811}{24.312} \times \frac{1}{2} \times (\quad) = 0.2223 \times (1.8293)$



RT $\xrightarrow{100°C/h}$ 1020°C $\xrightarrow{0.5}$ $\xrightarrow{40°C/h}$ 923 $\xrightarrow{f.c}$ RT

$(Ar + H_2)$ forming gas flow

EXHIBIT 2 for Sec. 1.131 Declaration
Patent Application No.: 09/885,471
Filed: June 20, 2001
Inventors: Sang-Wook Cheong et al

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[redacted] BR 145 NH93 MgB_2

(3/4 11) Room T Upper ram 3350
Lower ram 2200

T = 400°C UR 6100
LR 5500

T = 350°C UR 6200
LR 5500

RT $\xrightarrow[100°C/L]{}$ 400°C $\xrightarrow[100°C/L]{0.2L}$ 350°C $\xrightarrow[fc]{1L}$ RT

Final ~~60°C~~ UR ~~5550~~
LR 265C

~~[redacted]~~

BB

3/4/01 BB150 MgB_2 (Alfa Chemical)

(3/11/4) T = RT (UR 3000
LR 2000

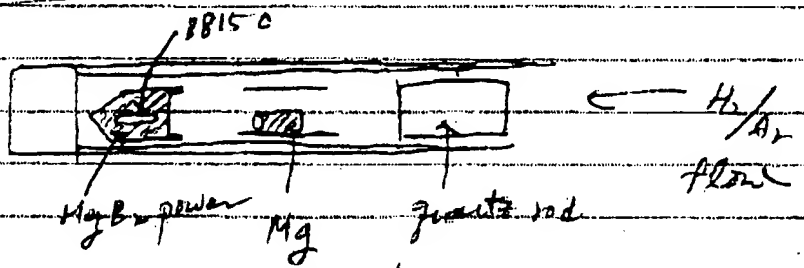
Ta ~~tube~~ capsule
gold
Ta T = 400°C (4000
3000

T = 650°C (4500
3000

final T = 40°C (4200
2100

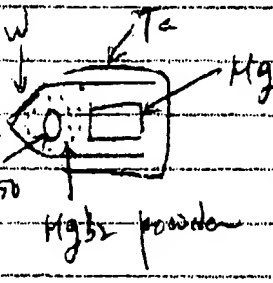
RT $\xrightarrow[1000°C/h]{}$ 400°C $\xrightarrow[1900°C/h]{0.2h}$ 660°C $\xrightarrow[f.c]{2h}$ RT

BB150a →



RT $\xrightarrow[100°C/h]{}$ 850°C $\xrightarrow[2h]{}$ RT $\xrightarrow[100°C/h]{}$

BB150b →



in quartz tube sealing

RT $\xrightarrow[200°C/h]{}$ 600°C $\xrightarrow[2h]{}$ 900°C $\xrightarrow[f.c]{}$ RT

EXHIBIT 4 for Sec. 1.131 Declaration
Patent Application No.: 09/885,471
Filed: June 20, 2001
Inventors: Sang-Wook Cheong et al